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MEASUREMENT OF BORON IN BORON-IRON BY DANGER COEFFICIENT METHOD

THOMAS E. CAREW
AND
DOUGLAS F. MOW

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Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE

IN

PHYSICS

United States Naval Postgraduate School
Monterey, California

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ABSTRACT

It has been proposed that the danger coefficient method be used with a low power research reactor to measure minute quantities of boron in iron-boron alloys. An investigation of the limits and accuracy of detection of boron in iron was undertaken using five iron-boron alloys containing different concentrations of boron. Samples of various sizes were introduced in turn into the AGN-201 reactor and the resulting changes in reactivity were determined. A change in reactivity of 4.62×10^{-8} per microgram of boron was found. The method is sensitive to a change in reactivity of $\pm 1.1 \times 10^{-6}$ for the AGN-201 reactor, giving a threshold of detection of 23.8 micrograms of boron under the experimental conditions employed. The relative error in the determination of boron content decreases with increasing sample size or boron concentration. The method offers a marked improvement in accuracy of measurement of low concentrations of boron-in-iron over that of conventional techniques. Extension of the methods employed here might be used for more complex ferrous alloys such as boron-steels.

The writers wish to acknowledge their indebtedness to Professors William W. Hawes and John R. Clark of the United States Naval Postgraduate School for their advice, assistance and encouragement, and the many hours of extra time which they devoted to this investigation.

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1. INTRODUCTION

When a substance is introduced into a reactor, it effectively changes the critical geometry. The resulting change in reactivity is a measure of the quantity of the substance introduced. The method of measuring the quantity of a substance through its effect on the reactivity of a reactor is known as the danger coefficient method.

The reactivity of a reactor operating at low power is measurably changed by the introduction of very small amounts of high neutron cross section substances, such as boron. This suggests that a low power research reactor may be useful in measuring the small concentrations of boron required for boron-steels. Boron-steels are found to have desirable hardenability properties if the weight percent of boron is held between .0005 and .0070. Concentrations greater than this result in brittleness and hot shortness[1]. Reliable measurements of such low concentrations cannot be made using conventional techniques such as spectrographic or chemical analysis[2]. Using the danger coefficient method with a low power research reactor offers promise of greatly improving the accuracy of this measurement.

Steels contain many impurities in addition to alloying elements. In order to determine the reactivity effect of boron in the presence of iron, uncomplicated by the presence of other elements, specimens of iron-boron relatively free of impurities were used for initial study.

[1] R.A. Grange, et al, Boron Calcium Columbium and Zirconium in Iron and Steel, John Wiley & Sons, New York, 1957, Part 1.

[2] E.C. Pigott, Ferrous Analysis Modern Practice and Theory, 2nd Ed. Rev., John Wiley & Sons, New York, 1953, pp. 108.

To evaluate the usefulness of a low power research reactor in the assay of boron in boron-iron an investigation was made of the experimental accuracy, the detection threshold for boron, and the specific reactivity change due to iron. It was also necessary to determine if the flux gradient near the center of the core would influence the results for samples of reasonably short length, and whether or not small samples would display significant neutron shielding.

2. THEORY

The reactor parameters applicable to the danger coefficient method are listed in this section. A brief discussion of the danger coefficient method is then given, followed by a discussion of the theoretical considerations imposed by the elemental composition of the test specimens used in this investigation.

a. Reactor Parameters[3]

The effective multiplication factor K_{eff} is a measure of the time rate of change of the neutron population. It may be defined as the number of neutrons available at the end of a neutron generation for each neutron present at the beginning of that generation, allowance having been made for losses due to escape and capture. When the quantity $K_{eff} - 1$ is a small positive number, the neutron flux increases exponentially with time. The stable reactor period is defined as the time required for the neutron flux to increase by a factor e:

$$\phi = \phi_o e^{t/T} \tag{2.1}$$

The reactivity (ρ) defined as $\rho = \frac{K_{eff}-1}{K_{eff}}$ is related to the period (T) through the inhour equation:

[3] S.Glasstone and M.C. Edlund, The Elements of Nuclear Reactor Theory, D. Van Nostrand Co., Inc., New York, 1952., Chap. 10.

$$(2.2) \qquad \rho = \frac{l}{T K_{eff}} + \gamma \sum_{i=1}^{6} \frac{A B_i}{1 + \frac{0.693\, T}{\tau_{\frac{1}{2}i}}}$$

The inhour equation is plotted with terms defined in Appendix A. The values of interest obtained from this equation are small numbers. It is convenient to employ the notation $\mu\rho$ for a value of equation (2.2) equal to 10^{-6}.

b. The Danger Coefficient Method

When a substance is introduced into a reactor, the resulting change in reactivity is due to the absorption, moderation, or elastic scatter of neutrons by interaction with the atomic nuclei of the substance. The reactivity change is proportional to the total macroscopic cross section of the substance. It is thus a measure of the quantity of the substance introduced.

The reactivity difference between a test specimen and a reference standard may be measured. If the reference standard is identical to the test specimen in all respects except for the absence of the element of interest, and if the specific reactivity change of the latter is known; then, a direct measure of the total amount of the element of interest may be obtained.

c. Theoretical Considerations.

Boron-iron specimens containing small amounts of impurities were used in this investigation as described in section 3. The impurities reported are carbon (0.06%), nitrogen (0.005%), and 0.10% other unspecified low neutron cross section substances. In the present purpose, the effect due to elastic scatter may be neglected as all samples were short in length and positioned in the center of the core where the reactivity change due to elastic scatter is small[4]. Thus, we need only

[4]Aerojet-General Nucleonics Staff, The AGN-201 Reactor Manual, AGN Memo 16, Operation of the Reactor, Aerojet-General Nucleonics, San Ramon, Calif. 1957.

consider the relative change in reactivity due to moderation and absorption. Absorption effects may be compared through the magnitude of the macroscopic cross section for absorption (Σ_a)[5]. Moderating effects may be compared through the relative magnitude of the slowing down power ($\xi \Sigma_s$)[6]. These quantities are listed in Table I below for one of the test specimens used.

TABLE I

1 Constituent	2 Weight (grams/gram of specimen)	3 Σ_a (barns)	4 ξ	5 $\xi\Sigma_s$
Iron	1.00	2.72×10^{22}	0.035	4.18×10^{21}
Boron	0.00025	1.05×10^{22}	0.174	9.65×10^{18}
Carbon	0.00060	9.60×10^{16}	0.158	2.28×10^{19}
Nitrogen	0.00005	4.04×10^{18}	0.136	2.87×10^{18}

The values in column 3 are a measure of the probability per gram of specimen of neutron absorption by the respective nuclei. The values in column 5 of the table are a measure of the probability per gram of specimen of an effective inelastic collision of a neutron with the respective atomic nuclei. The effect on the reactivity of the reactor caused by a single neutron absorption is several orders of magnitude greater than the reactivity effect of an average moderating collision[7]. First considering only the iron and boron it is thus seen from the values in columns 3 and 5 that the change in reactivity due to moderation for each is insignificant compared to the change in reactivity due to absorption. Iron and boron will therefore be treated as pure absorbers. It

[5,6,7] S. Glasstone and M.C. Edlund, op cit.

may also be seen that the reactivity change due to the small concentrations of carbon and nitrogen in the test specimens is insignificant for either process compared to the effects due to iron and boron. The presence of these impurities may therefore be ignored.

We now will consider briefly the possible effect on reactivity of the 0.10% of other unspecified impurities.

Many impurities which are normally found in iron have neutron cross sections close to that of iron so their effect in small concentrations could not be distinguished from that of iron. Manganese, normally present in iron, exhibits high cross section absorption peaks in neutron energy ranges above the thermal[8]. Its effect cannot therefore be predicted by considering only its thermal neutron cross section. The presence of manganese in trace amounts in the specimens is probable. If the concentration were known, its effect could be experimentally determined and a correction made for its presence. In this investigation we have assumed that manganese, and other elements of similar effect were not present in concentrations large enough to significantly affect our results. The experimental result found for the change in reactivity due to neutron absorption for iron will be in error to the extent that this assumption is not completely valid. The relative assay for boron, however, will remain unchanged if the manganese concentration is constant for all the test specimens.

[8]D.J. Hughes and J.A. Harvey, Neutron Cross Sections, Vol. 5 of Selected Reference Material, U.S. Atomic Energy Program, Associated Universities, In., 1955.

3. PROCEDURE

Five specimens of iron, free of significant impurities, were furnished by the Scientific Laboratory of the Ford Motor Company in the form of rods of approximately $\frac{1}{2}$ inch diameter. Four of the specimens contained different concentrations of boron as determined by spectrographic analysis. The analyses furnished by the Ford Motor Company are given in Table II.

TABLE II

Specimen	Heat Number	Boron Content (Weight %)
A	396	.0001
B	383	.0020
C	387	.0050
D	385	.0072
E	458	.0250

Note: All specimens contain .06% carbon, .005% nitrogen and .10% other low neutron cross section impurities.

The specimens were machined, cut into nominal lengths of 1/16, 1/8, 1/4, and 1/2 inches. They were then mounted in polyethylene sleeves as shown in Fig. 1. This permitted flexibility in the introduction of any desired sample size in multiples of 1/16 inch.

The AGN-201 reactor[9] operating at a maximum power of 0.1 watt was used in this investigation. The neutron flux at the center of the core is 4.5×10^6 neutrons/cm^2-sec at maximum power. Fig. 2 shows a view of the reactor and control console. A cross sectional view of the reactor is in Fig. 1, together with an enlarged view of the core tank, and a detailed view of the control rod assembly.

[9] A.T. Biehl, et al, Compact Low Cost Reactor Emphasizes Safety, Nucleonics, 14, 100-103, Sep 1956.

In the reactor, the neutron flux is monitored by a BF_3 detector mounted in the water jacket just outside of the lead shield. The output of the detector is proportional to the neutron flux and is thus a measure of the power level. The output is amplified at the control console and fed into a Varian strip recorder, Model G-10, where a permanent record is obtained of flux level, or power, as a function of time. When the reactor is increasing in power, the stable reactor period may be obtained from the flux-time record of the recorder by use of the exponential relationship.

(3.1) $$T = \frac{(t - t_o)}{(\ln \phi - \ln \phi_o)}$$

Test specimens were mounted in the sample holder shown in Fig. 1 and positioned in the glory hole so that the specimen was at the center of the core. Polyethylene, graphite, and lead plugs were added to match the core, reflector, and shielding in order to achieve a symmetrical configuration.

With the reactor at a low power level, the sample holder containing the test specimens was positioned in the glory hole and the control rods were adjusted to give a convenient positive period. A record of the resulting change in neutron flux as a function of time was obtained. When the power level reached the designed limit of 0.1 watts, the sample holder was withdrawn from the glory hole causing the power to decrease due to the loss of the moderating effect of the polyethylene in the holder. During the time required for the power level to fall, the test specimen in the holder was changed. When the power level fell to a pre-selected level the holder was reinserted. The next measurement was thus obtained from the resulting power rise without readjusting the control rods. This procedure was repeated to obtain a sequence of measurements. The period was determined from the recorder plot through use of equation

Figure 1 - Specimens & Equipment & Reactor

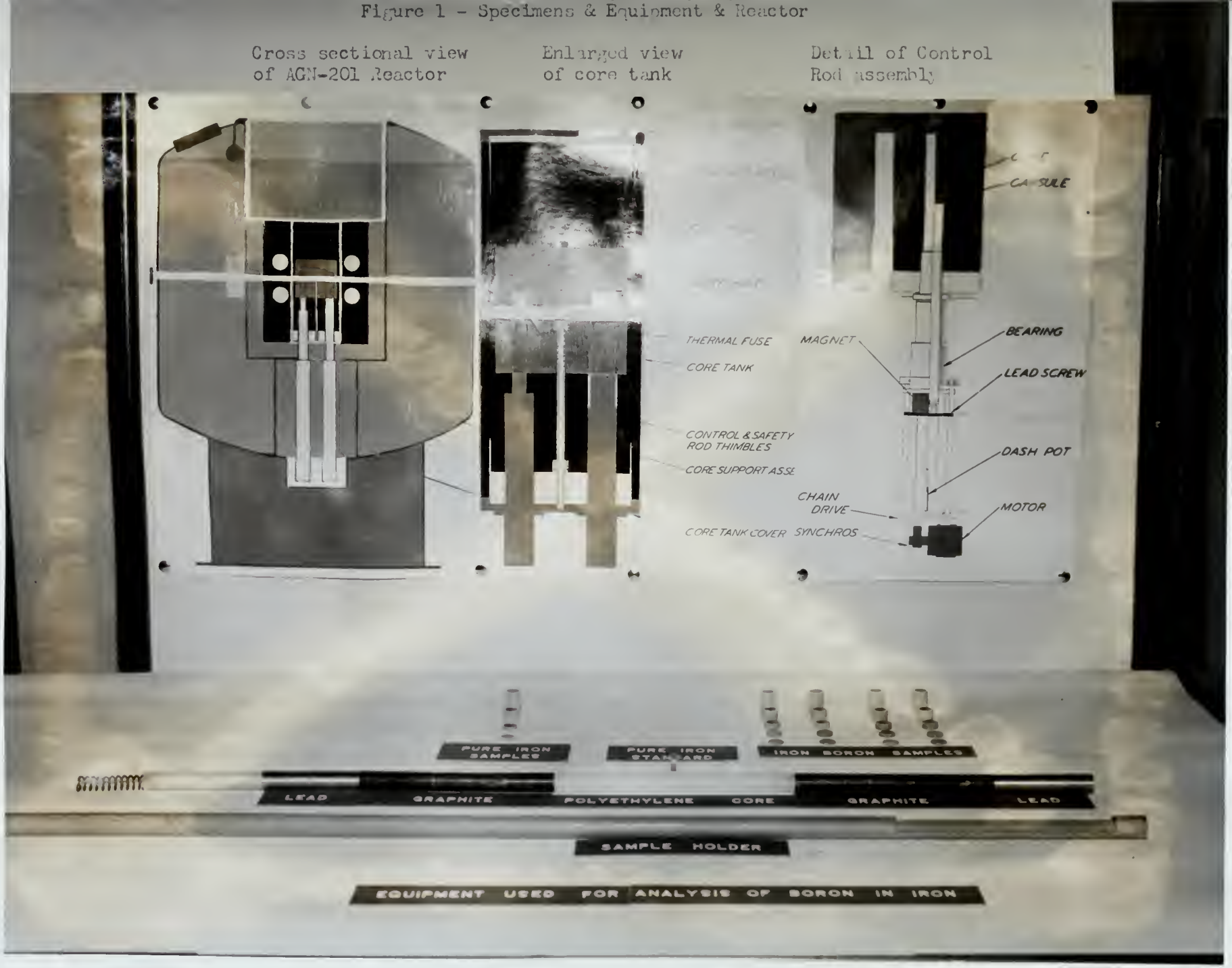


Fig. 1

Figure 2

AGN-201 Reactor and Control Console

(3.1). With the value of T known, the corresponding value of reactivity was obtained from an enlarged plot of the inhour equation (Appendix A). This procedure is essentially the same as that reported by Branson and Jensen.[10]

One of the prepared samples was selected as a reference standard, and the measured changes in reactivity of all others were taken with respect to it. The $\frac{1}{4}$ inch sample of boron-free iron (Specimen A) was selected as suitable for this purpose.

The sample to be measured and the reference standard were alternately introduced into the reactor until five or six determinations were made of each. A sequence of such measurements is shown in Fig. 3. The downward drift of the data, seen in Fig. 3, is typical of that found in all of the measurements taken. This drift was also reported by Branson and Jesson[11]. It was nearly constant during a sequence of measurements. The cause of the drift was not investigated; its effect on the measurements, however, was canceled by measuring all test specimens against a reference standard. The repeated measurements were made in order to evaluate the statistical fluctuations and to correct for the drift. A sequence of such measurements required about one and a half hours to complete.

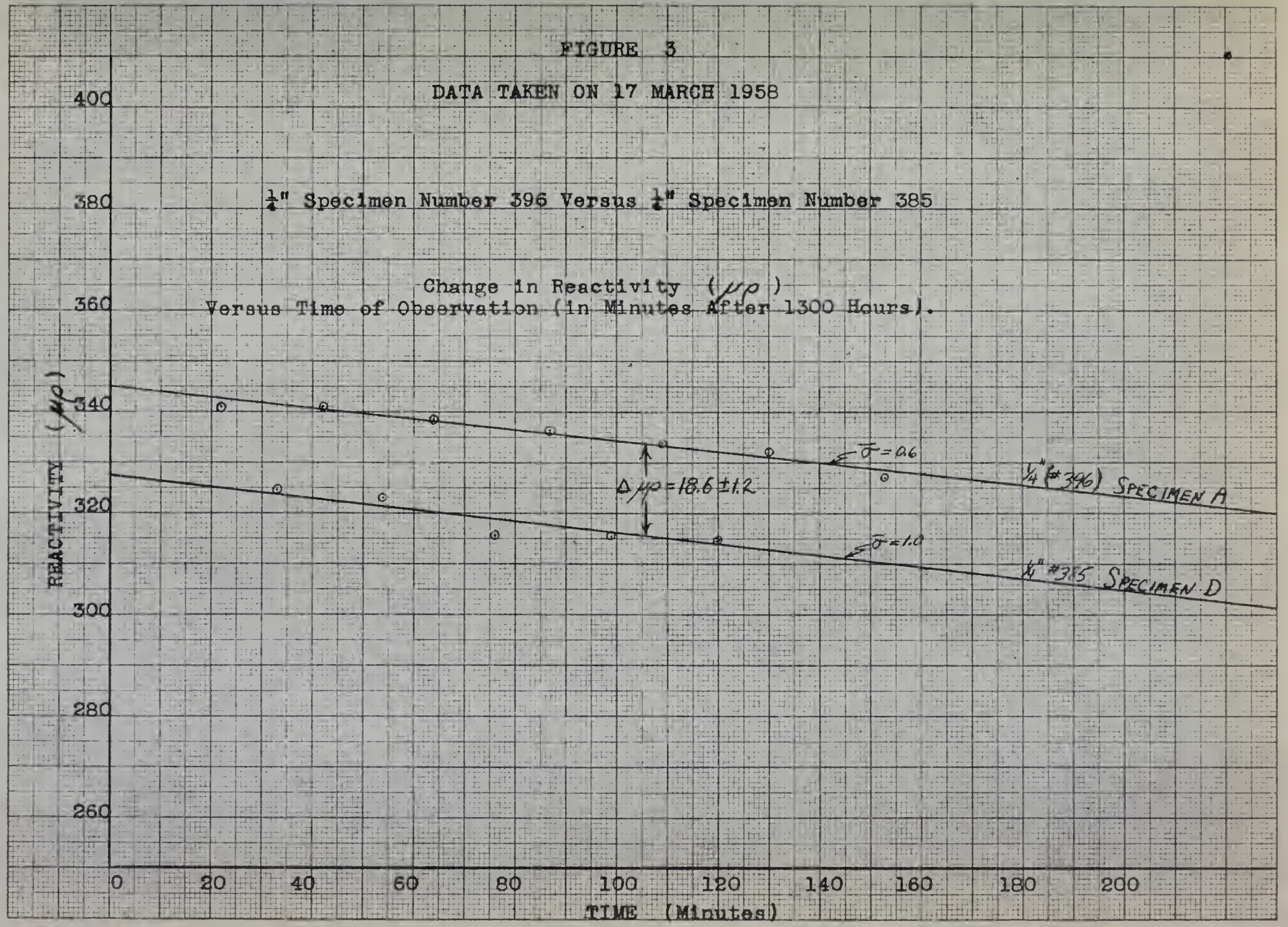
At the start of a typical sequence of measurements, the control rods were positioned to give a positive period of from 200 to 300 seconds with the reference standard in place in the core. At the same control rod setting, introducing the test specimen generally resulted in a longer period. Reliable measurements were made with period differences up to

[10] J.J. Branson, Jr. and G.E. Jessen, Estimation of Boron Impurities in Graphite by Danger Coefficient Method, Thesis, U.S. Naval Postgraduate School, Monterey, Calif., 1958.

[11] J.J. Branson, Jr. and G.E. Jessen, op cit.

FIGURE 3

DATA TAKEN ON 17 MARCH 1958



600 seconds. Large period differences were avoided in the measurements taken by introducing intermediate standards for those measurements where the period difference would exceed 600 seconds.

4. TREATMENT OF DATA

The difference in reactivity with which we are concerned is the constant difference between the two lines as shown in Fig. 3. A best fit of the data was made by the method of least squares[12]. The drift was the same for both the reference standard and the test sample. The standard deviations were computed for each line, from which the standard deviation of the difference measurement was determined.

A number of measurement sequences were made from samples of various sizes from the same specimen. Results are given in Table III. The change in reactivity as a function of sample weight for each specimen is plotted in Fig. 4 and 5. The slopes of the lines represent the average change in reactivity per gram of specimen.

In the experimental procedure employed, a sample was substituted in the sample holder for an equal volume of polyethylene. Weight measurements gave a value of 0.109 grams of polyethylene removed for each gram of specimen added. The reactivity change of polyethylene is 12.8 $\mu\rho$/gram at the center of the core[13]. This value was confirmed by independent measurements made by the authors. This leads to the result that there is a change in reactivity of 1.4 $\mu\rho$/gram of specimen added due to the removal of an equal volume of polyethylene.

[12] P.G. Hoel, Introduction to Mathematical Statistics, John Wiley & Sons, Inc., 1947.

[13] AGN Memo 16, op cit.

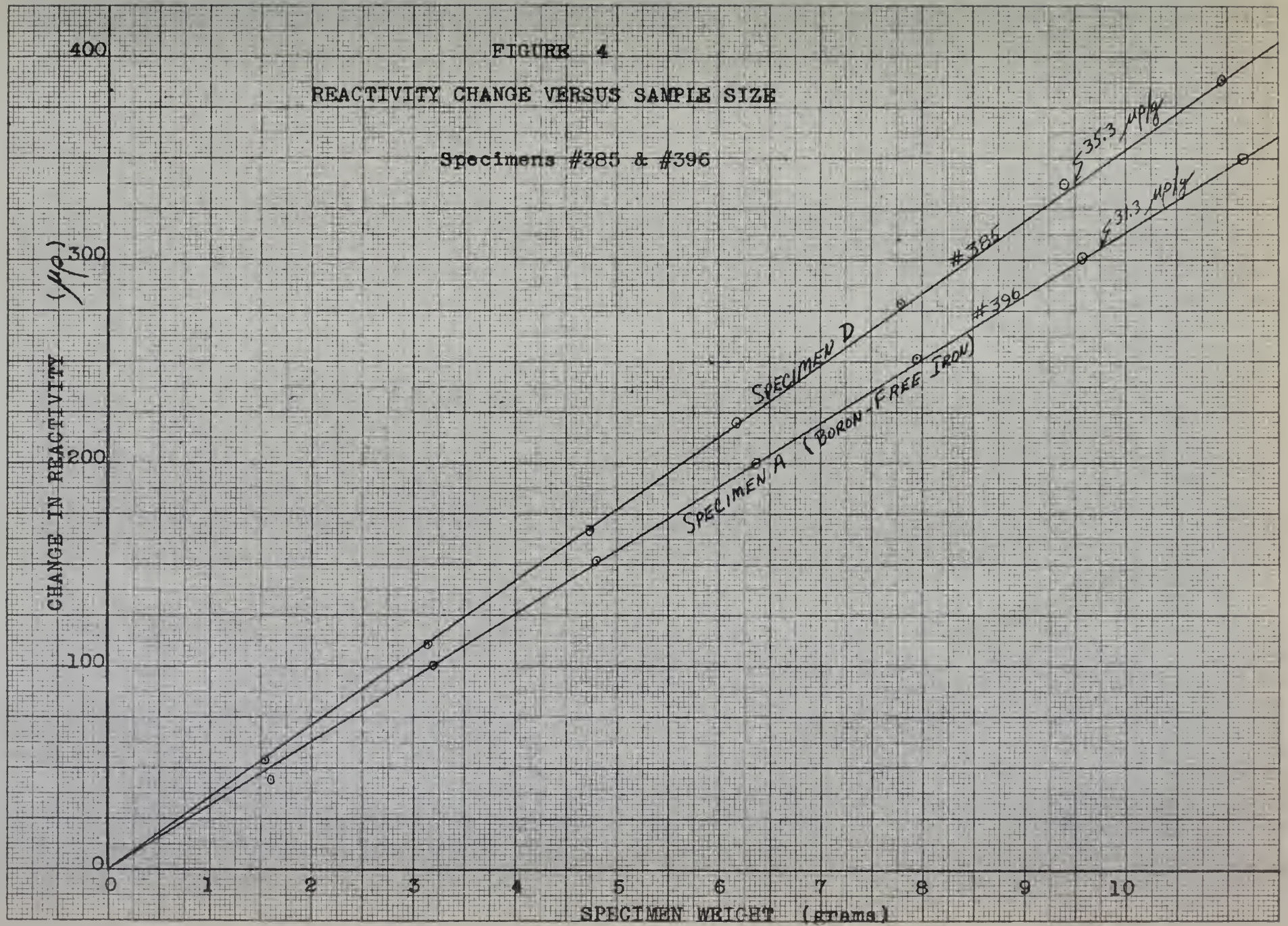
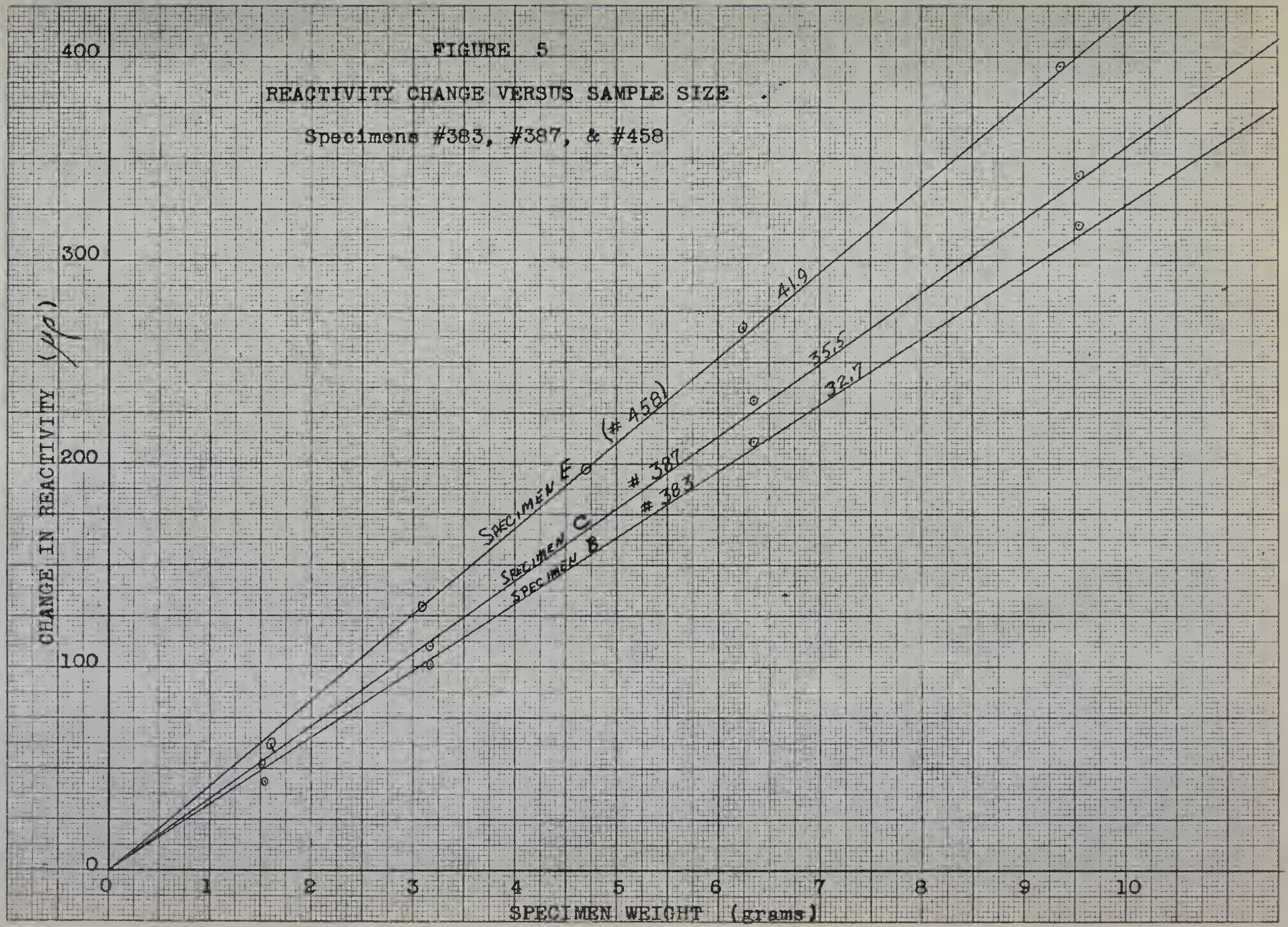


FIGURE 4
REACTIVITY CHANGE VERSUS SAMPLE SIZE
Specimens #385 & #396
CHANGE IN REACTIVITY (μρ)
SPECIMEN WEIGHT (grams)
0
100
200
300
400
0
1
2
3
4
5
6
7
8
9
10
SPECIMEN D
#385
35.3 μρ/g
SPECIMEN A (BORON-FREE IRON)
#396
31.3 μρ/g

FIGURE 5

REACTIVITY CHANGE VERSUS SAMPLE SIZE

Specimens #383, #387, & #458



The slope of the line for specimen A, given in Fig. 4, is 31.3 $\mu\rho$/gram. Subtracting 1.4 $\mu\rho$/gram for the effect due to the removal of polyethylene gives a value of 29.9 $\mu\rho$/gram for the specific reactivity of iron.

TABLE III

SPECIFICATIONS AND DATA

Specimen	Nominal Length (inches)	Weight (grams)	Reactivity Change ($\mu\rho$)
A	1/16	1.5876	44.0 ± 1.9
	1/8	3.1985	100.0 ± 1.4
	3/16	4.7861	150.0 ± 0.9
	1/4	6.3810	200.0 ± 0.0
	5/16	7.9686	252.0 ± 0.7
	3/8	9.5795	302.0 ± 0.9
	7/16	11.1671	350.0 ± 1.3
	1/2	12.7680	401.0 ± 2.3
B	1/16	1.5612	43.7 ± 2.0
	1/8	3.1839	101.0 ± 1.3
	1/4	6.3510	210.5 ± 0.9
	3/8	9.5349	318.0 ± 1.9
	1/2	12.7235	416.7 ± 1.5
C	1/16	1.5300	51.5 ± 2.1
	1/8	3.1750	109.0 ± 1.0
	1/4	6.3700	232.0 ± 0.9
	3/8	9.5450	341.8 ± 1.4
	1/2	12.7285	449.3 ± 1.6
D	1/16	1.5750	53.0 ± 1.9
	1/8	3.1670	110.0 ± 0.9
	3/16	4.7420	167.2 ± 1.6
	1/4	6.2388	218.6 ± 1.2
	5/16	7.8138	280.0 ± 1.6
	3/8	9.4058	337.3 ± 1.7
	7/16	10.9808	390.8 ± 1.6
	1/2	12.7693	446.5 ± 1.5
E	1/16	1.6040	61.4 ± 1.0
	1/8	3.1101	129.0 ± 1.6
	3/16	4.7141	198.2 ± 0.9
	1/4	6.2610	267.0 ± 0.9
	3/8	9.3711	394.5 ± 1.1
	1/2	12.7512	529.0 ± 1.3
	9/16	14.3552	590.8 ± 1.8
	5/8	15.8613	644.0 ± 1.4

Notes:
1. All specimens are cylindrical with diameters 0.5 inches.
2. All reactivity measurements based on difference from the 1/4 inch specimen A.
3. A determination of the mean standard deviation for more than forty observations resulted in a value of ± 1.1 $\mu\rho$.

This reactivity change may be attributed entirely to the neutron absorption of iron.

Since the specimens differed from each other only in boron content, the slope differences in Fig. 4 and 5 are due only to the differences in boron concentration. These slope differences provide an accurate measure of the relative boron concentration of the five specimens. In order to determine absolute values, however, it is necessary to know the change in reactivity per gram of boron at the center of the core. This can be determined experimentally, or a value may be calculated using a semi-empirical approach. The approach used in this investigation was to relate the macroscopic cross sections for absorption of boron and iron to a corresponding change in reactivity.

The microscopic thermal neutron cross section for absorption for iron is taken as 2.50 barns. The macroscopic cross section per gram of iron is then given by:

$$(4.1) \qquad \Sigma_a)_{Fe} = \frac{(1)\,(N_o)\,(2.50 \times 10^{-24})}{A} = 2.72 \times 10^{-2} \text{ cm}^2/\text{gram of iron}$$

The macroscopic thermal neutron cross section for absorption per gram of boron is:

$$(4.2) \qquad \Sigma_a)_B = \frac{(1)\,(N_o)\,(755 \times 10^{-24})}{A} = 42 \text{ cm}^2/\text{gram of boron}$$

where N_o = Avogadros No.

A = Atomic Weight

For a first approximation we will take the ratio between the reactivity change per gram of boron and the reactivity change per gram of iron as equal to the ratio of the macroscopic thermal neutron cross sections per gram of boron and iron, i.e.,:

$$(4.3) \qquad \Delta\rho_B = \frac{\text{Reactivity change}}{\text{gram of Iron}} \quad \frac{\Sigma_a B}{\Sigma_a Fe}$$

Substituting for the known values on the right side of this equation gives the desired quantity:

(4.4) $$\Delta\rho/\rho_B = (29.9)\,\frac{42}{2.72 \times 10^{-2}} = 4.62 \times 10^4\ \rho/\text{gram of Boron}$$

This quantity is more conveniently expressed in terms of μgm of boron, i.e.: $4.62 \times 10^{-2}\ \rho/\mu\text{gm}$.

With the above value for the change in reactivity per microgram of boron applied to the slope differences in Fig. 4 and 5, and assay of boron concentration for each test specimen is obtained. For example, the slope difference between specimen A and specimen E is 10.6 ρ/gram. The assay for specimen E is then:

(4.5) $$10.6\ \rho \times \frac{1}{4.62 \times 10^{-2}\ \rho/\mu\text{gm of boron}} = 230\ \mu\text{gm of Boron/gram}$$

5. RESULTS

Assays of the specimens examined for boron concentration are given in Table IV.

TABLE IV

Specimen	Reported assay(boron) (p.p.m.)	Experimental Assay(boron) (p.p.m.)	Percent error (standard dev.)
A	1	----	----
B	20	30.3	6.2%
C	50	91.0	2.1%
D	72	86.7	2.2%
E	250	230.0	0.8%

The measured differences between the sample and the reference standard were, on the average, determined with a precision of $\pm$ 1.1 ρ as is shown in Table III basic data. From equation (4.4) the change in reactivity due to boron is found to be $4.62 \times 10^{-2}\ \rho/\mu\text{gm}$. The average

error in the detection of total boron under the experimental conditions of this investigation is thus:

$$(5.1) \qquad \pm 1.1 \times \frac{1}{.0462\ \mu\rho/\text{gm}} = \pm 23.8 \text{ gm. of Boron}$$

It may be seen that a 23.8 gram sample of boron-iron containing 1 p.p.m. of boron will result in a change in reactivity of 1.1 $\mu\rho$ when compared to a boron-free iron standard of similar size. This is reasonably the threshold of detection for the AGN-201 reactor using small samples at the center of the core.

6. DISCUSSION

Table IV compares the boron content based on spectrographic analysis with that we obtain. Spectrographic analysis is subject to considerable error in measuring low concentrations. It is seen however, that the quantities reported are in substantial agreement with those found by the danger coefficient method. The percentage error listed is based entirely on the experimental accuracy. The assay may be subject to additional error to the extent that the presence of significant amounts of impurities having high energy absorption resonances will change the basic value of 29.9 $\mu\rho$/gram attributed to iron; and to the extent that the changes in reactivity are not directly proportional to the respective macroscopic absorption cross sections.

In all instances, the iron-boron specimens displayed a somewhat greater degree of scatter than the boron-free specimen (Fig. 4 and 5). This may be due to an inhomogeneity in the distribution of the boron phase in the iron. An attempt was made to identify the boron phase and evaluate its distribution by microscopic examination of polished and etched specimens. The results were inconclusive, however, due to the difficulty in identifying the small amounts of boron involved in the presence of larger amounts of carbon.

An attempt was made to measure the reactivity change between the reference standard and a plain polyethylene core. This was expected to result in a value of 200 $\mu\rho$ as shown by the plot in Fig. 4. The actual measurement, however, was 219 $\mu\rho$, a deviation greater than can be attributed to measurement error. Differences in periods here were in excess of the 600 second maximum.

The change in reactivity with increasing sample size was found to be linear throughout the entire range of samples used. The largest sample represented a total macroscopic cross section of 0.597 cm^2, which is approximately equal to 22 grams of iron. The plots of change in reactivity versus sample size given in Fig. 4 and 5 show no departure from linearity. This indicates that neutron shielding is not important in samples up to the maximum studied and the flux distribution through the sample was essentially constant. The possibility exists then, that still larger sample sizes could be employed to attain greater accuracy.

7. CONCLUSIONS

Low Boron concentration in iron can be determined with significantly greater accuracy by the danger coefficient method than by spectroscopic analysis. The detection threshold of boron in iron for small samples at the center of the core is 23.8 micrograms for the AGN-201 reactor. Reactivity changes may be found with an average error of $\pm$ 1.1 $\mu\rho$ under the experimental conditions employed.

8. ACKNOWLEDGMENTS

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APPENDIX A

THE INHOUR EQUATION

$$\rho = \frac{l}{T K_{eff}} + \gamma A \sum_{i=1}^{6} \frac{B_i}{1 + \frac{0.693\,T}{\tau_{\frac{1}{2}i}}}$$

ρ = reactivity

l = mean lifetime of thermal neutrons

T = stable reactor period

K_{eff} = effective multiplication constant

γ = mean effectiveness of delayed neutrons

A = the delayed fraction of total fission neutrons

B_i = fraction of delayed neutrons in the ith group

$\tau_{\frac{1}{2}i}$ = half life of the ith group precursor

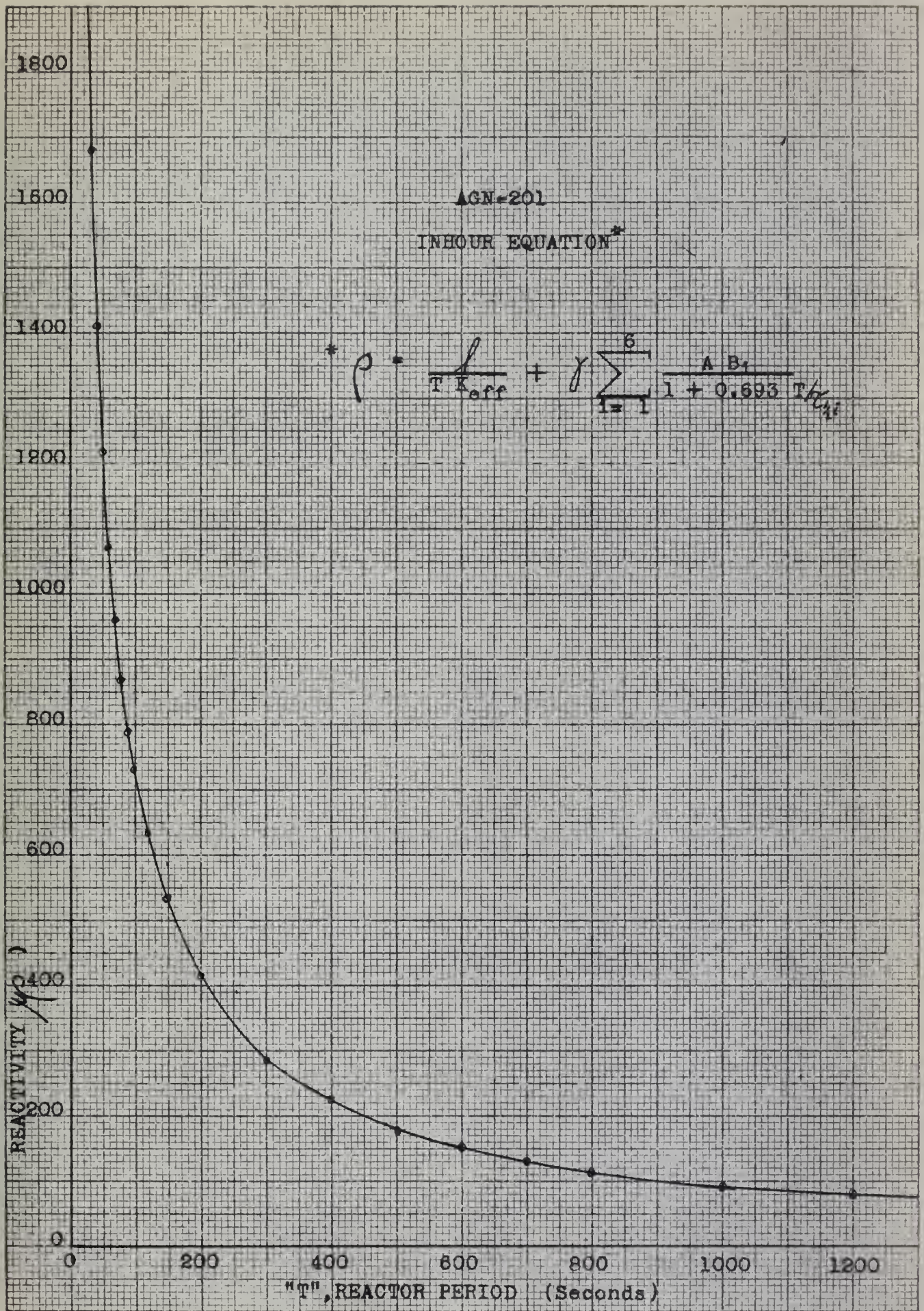
SPECIFIC VALUES

γ = 1.14 (that is, the delayed neutrons are emitted at a lower energy than prompt neutrons and are therefore more effective)

A = 0.00665

l = 10^{-4} sec

Group	B_i	$\tau_{\frac{1}{2}i}$ (sec)
i = 1	.038	54.51
2	.213	21.84
3	.188	6.00
4	.407	2.23
5	.128	0.496
6	.026	0.179


AGN-201
INHOUR EQUATION*
$\rho = \frac{\ell}{T K_{eff}} + \gamma \sum_{i=1}^{6} \frac{A B_i}{1 + 0.693 T/t_{1/2 i}}$
1800
1600
1400
1200
1000
800
600
400
200
0
REACTIVITY (ρ)
0
200
400
600
800
1000
1200
"T", REACTOR PERIOD (Seconds)

thesC2
Measurement of boron in boron-iron by da
3 2768 002 08519 3
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